

Metal Quantifications by TXRF in Solution with Large Matrices

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Total Reflection X-ray Fluorescence (TXRF) is an excellent analytical technique for simultaneous multi-element quantification.¹ It has several advantages in comparison with other analytical techniques such as a fast and easy sample preparation procedure with a limited amount of chemicals, no need for new calibration curves each new series of measurements, short process times and low operational costs. The main advantages of TXRF in comparison with common XRF are less matrix effects and lower detection limits.

The research activities of The Laboratory of Inorganic Chemistry (KU Leuven – University of Leuven, Belgium) can be described by three keywords: (1) ionic liquids, (2) hydrometallurgy, and (3) rare earths. The groups focusses on the development of hydrometallurgic separations methods, often based in ionic liquid technology, for separating metals.

On average, elemental concentrations in more than 500 liquid samples are measured in our lab every week by three TXRF machines having a molybdenum X-ray source (S2 Picofox, Bruker). The matrices and metals present in the solution are often very different. Frequently, diverse problems are faced such as overlapping peaks between one element in a large concentration and one element in a small concentration, matrix effects and significant reduced counts rates by large salt concentrations in the aqueous phase (e.g. 10 M NH_4NO_3 , CaCl_2 , ...), inhomogeneous sample distribution due to fractional crystallization during the drying procedure, improper determination of halides due to the volatility of HX compounds,^{2,3} quantification problems or yttrium for which the secondary X-rays are overlapping with Raleigh and Compton scattering of the Mo source, and measurements in the organic, non-volatile and oil-like ionic liquid matrices.

In this talk, our experience related to several measurement problems will be shared and solutions will be given where possible.

- (1) R. Klockenkämper and A. von Bohlen, *Total-Reflection X-Ray Fluorescence Analysis and Related Methods*, John Wiley & Sons, 2014.
- (2) T. Vander Hoogerstraete, S. Jamar, S. Wellens, and K. Binnemans, *Anal. Chem.*, 2014, **86**, 1391-1394.
- (3) T. Vander Hoogerstraete, S. Jamar, S. Wellens, and K. Binnemans, *Anal. Chem.*, 2014, **86**, 3931-3938.